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<b>(21) International Application Number:</b> PCT/EP96/05616 <b>(22) International Filing Date:</b> 13 December 1996 (13.12.96) <b>(30) Priority Data:</b> 9525641.8 15 December 1995 (15.12.95) GB <b>(71) Applicant (for all designated States except US):</b> ROCKWOOL INTERNATIONAL A/S [DK/DK]; Hovedgaden 584, DK-2640 Hedehusene (DK). <b>(72) Inventors; and</b> <b>(75) Inventors/Applicants (for US only):</b> KRAGLUND, Arne [DK/DK]; Hjortekaersvej 151 C, DK-2800 Lyngby (DK). VISLER, Torben [DK/DK]; Hestkobvej 9B, DK-3460 Birkerød (DK). RANLOV, Jens [DK/DK]; Puggaardsgade 10, 3th., DK-1573 København (DK). CHRISTENSEN, Vermund, Rust [DK/DK]; Soager 11, St. Valby, DK-4000 Roskilde (DK). <b>(74) Agent:</b> GILL JENNINGS & EVERY; Broadgate House, 7 Eldon Street, London EC2M 7LH (GB).		<b>(81) Designated States:</b> AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, HU, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, TJ, TM, TR, TT, UA, UG, US, UZ, VN, ARIPO patent (KE, LS, MW, SD, SZ, UG), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).  <b>Published</b> <i>With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i>
<b>(54) Title:</b> PRODUCTION OF MINERAL FIBRES  <b>(57) Abstract</b>  MMV fibres containing high amounts of Al <sub>2</sub> O <sub>3</sub> are made from a melt which is formed by melting a charge which includes briquettes formed from alumina sand which is contaminated with foundry residues.		

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### PRODUCTION OF MINERAL FIBRES

This invention relates to the production of Man-Made Vitreous Fibres (MMVF) that have a relative high content of aluminium, and in particular it relates to the production of such fibres which are biologically soluble, that it is to say they have an acceptable rate of biologically useful degradation when tested in an appropriate liquid.

It is well known to form in a furnace, such as an electric furnace, tank furnace, shaft furnace or a cupola furnace, a mineral melt and to use this melt for various industrial purposes. The mineral melt is generally formed from a blend of minerals selected so that they produce a melt having the desired melting point and other properties, having regard to the intended end use. The mineral materials are usually freshly produced or mined materials such as rock or slag (often after crushing) and sand.

In EP-A-508589 it is proposed to form a melt from a plurality of solid waste streams that are used in proportions such that the melt has a content within certain defined ranges. Materials that are mentioned for use as this plurality of waste streams are municipal incinerator bottom ash, hazardous wastes incinerator bottom ash, baghouse or precipitator dust, steel plant dust, electroplating sludge, electrochemical machining sludge, waste foundry sands, contaminated soils, dried and contaminated sewage solids, cementitious fixation, coal combustion fly ash, inorganic paint pigment residues and spent refractory materials. The furnace can be a coke-fired cupola furnace of the type used in the grey iron casting industry. The melt is drained from the furnace under conditions that allow separation of free metal from the solution of oxides, which can be poured into moulds or quenched.

In JP-A-55140725 mine slime and/or waste enamel and/or waste moulding sand are blended into steel making slag and the resultant blend is melted in an electric furnace and converted into fibres.

Traditionally MMV fibres were made from materials that were economically and geographically convenient to use. However it is now recognised that the solubility of MMV fibres and other properties is very dependent on their chemical analysis, and thus on the analysis of the melt from which the fibres are formed. Accordingly there is need to be able to select the analysis of the melt accurately, having regard to the particular properties that are required. Accordingly it is generally no longer appropriate to use merely those raw materials which are geographically and economically convenient to the plant where the fibres are being made and, instead, it is increasingly necessary to select particular materials having regard to the analysis and properties required of the fibres.

It is common for some or all of the mineral material which is to be incorporated in the melt to be provided in the form of moulded briquettes, and in particular fine material such as sand is usually incorporated into briquettes.

The need to use raw materials selected according to the desired chemical analysis rather than according to economic convenience means that there is a tendency towards an increase in the cost of the raw materials that are used for making these carefully defined MMV fibres. Cost is a particular problem when the desired final blend has an analysis which tends to require materials which are rather expensive. For instance, we have described in W095/34516 that fibres having a very low content of alumina can be made using silica sand which can be recovered foundry sand. However a particular problem arises when it is desired to make fibres having high alumina content, namely above about 14% by weight and often above 18% or 20% by weight.

Throughout this specification all analyses are quoted by weight of the oxides.

It might be thought that it would be economic and convenient to provide such materials using a charge which

contains a predetermined amount of a raw material which has high alumina content. For instance the inclusion of alumina sand of the type frequently known as calcined bauxite might appear appropriate. Unfortunately these high alumina raw materials tend to be unavailable in many areas or to be very expensive or both. This places undesirable constraints on the selection of materials that can be used for the manufacture of high alumina MMV fibres.

In the invention, MMV fibres are made by a method comprising forming moulded briquettes of particulate mineral material, forming a melt by melting in a furnace a mineral charge comprising the briquettes and forming fibres from the melt, and in this method the melt and the fibres have a content, measured as oxides, that includes alumina in an amount of above 14% by weight and the briquettes are formed of particulate mineral material that includes used alumina sand contaminated with foundry residues.

The melt and the fibres generally contain  $\text{Al}_2\text{O}_3$  in an amount of at least 18 or 19%, and often at least 20%. Thus the amount may be above 25% and as high as 30% or more, for instance up to 36 or 38% or higher.

By referring to "alumina sand" we mean any fine particulate inorganic material which can be melted into a melt from which MMV fibres can be made and which has a relatively high alumina content, for instance at least 20% and usually at least 30% or 40% in weight. Preferably it has at least 50% by weight alumina and it can have an alumina content as high as 80% or more.

We have realised that it is satisfactory, and indeed very desirable, to make high alumina MMV fibres from a charge containing briquettes which have been formed utilising alumina sand which is contaminated with foundry residues. As a result it is now possible to use a single batch of alumina sand for two separate processes, thereby gaining significant economic advantages, without incurring any technical disadvantages in either process.

Alumina sand is much too expensive to be used in conventional foundry processes in competition with traditional silica sand. However it does have certain properties that make it very attractive, from a technical point of view, for certain foundry processes. Unfortunately the cost of buying fresh alumina sand and then dumping it after foundry use is a severe deterrent from developing these processes. In the invention, we use the alumina sand for the foundry processes and then we use the resultant contaminated alumina sand for making MMV fibres.

Thus, by the invention, we simultaneously make the foundry process economically viable and provide an economically acceptable source of alumina for the production of high alumina MMV fibres.

The foundry residues are residues that accumulate in the sand when it is used for foundry casting. Thus the used sand will normally be contaminated with residues of binder, such as phenol formaldehyde resin, furan, bentonite or other foundry binder from the casting operation. It may also contain sand fines having a smaller size than the sand would normally have. It may contain residues of metallurgical fines, metal or metal compounds derived from the casting operation. The cast metal can be iron, leading to iron or iron oxide residues. When aluminium is the cast metal, the residues may be of aluminium or aluminium oxide.

Due to the presence of these various residues, the used sand is usually considered unsuitable for any useful process unless it is first subjected to thermo/mechanical regeneration process comprising the steps of crushing, sieving, washing and incinerating to remove the contaminants and fines. However such reclamation processes tend to be very expensive and so render the use of the reclaimed sand uneconomic. For instance such a process is described in Mineral Processing No.8, August 1987, pages 456 to 462 by Bauer, where the significant process stages

are magnetic separation fluid bed treatment and counter flow baffling.

In the invention, the contaminated sand is used without significant prior reclamation processes. Indeed, 5 if any reclamation process is conducted, it is usually confined to sieving the sand.

Thus the invention simultaneously solves two problems, namely the provision of an economic source of alumina for high alumina MMV fibres, and a utility for the used sand 10 which will give the used sand sufficient value to render economic its use in the specialised foundry processes.

One advantage of the invention lies in the fact that it is not necessary to remove fines from the contaminated alumina sand before it may be used. As explained above, 15 this was previously always considered necessary if the alumina sand was to be useful. Thus particularly economical use of contaminated alumina sand is made possible by the invention.

In fact, the presence of fines can be advantageous in 20 the invention. Fines exhibit excellent melting and dissolution properties in conventionally used melting processes. This is due to their need for shorter residence times at any given melting temperature than larger particle size alumina sand.

25 This feature means that it is possible to use higher amounts of high melting point materials such as the contaminated alumina sand in the charge than would otherwise be possible. The presence of fine particle size alumina sand also minimises or eliminates the need to 30 incorporate fluxing agents, and thus avoids the constraints they place on the choice of chemical compositions.

The amount of contaminated alumina sand is preferably at least 5%, usually at least 10% or 20% based on the total mineral charge and often at least 30%. Usually it is not 35 more than 60%, or sometimes 70 or 75%, and generally it is in the range 20 to 40 or 45% by weight of the total mineral charge.

Suitable alumina sands are calcined bauxite, chamotte, andalusite, kyanite, plagioclase, silimanite, mullite and kaolin and fused alumina. Fused spinel is another suitable high  $\text{Al}_2\text{O}_3$  material.

5       The use of various high alumina sands in foundry processes is described in the literature, for instance in Heat Transfer of Various Moulding Materials, Locke et al, Trans.Am.Foundrymans Soc., 1954, volume 62, pages 589-600.

10       Although it is possible for the briquettes which contain the used alumina sand to consist (as regards the inorganic components) solely of that sand, briquettes are usually formed of a blend of at least 10% by weight of the used sand with at least 10% by weight of other inorganic material. Often the amount of used alumina sand is at  
15       least 30% and frequently at least 50% or 60% by weight of the inorganic component of the briquettes. The mix from which the briquettes are formed may be a blend of the used alumina sand with virgin inorganic sand or rock but often the briquettes are formed from a blend of the used alumina  
20       sand with other inorganic material which includes industrial waste material. Suitable industrial waste materials include converter slag or other slag from steel making processes, glass, mineral fibre cement, power plant ash, wood ash and steel plant dust and MMV fibre products,  
25       for instance bonded MMV fibrous material. This fibrous material may be recycled from the process or may be waste, previously manufactured, material.

When it is desired to increase the silica content, it can be convenient to incorporate in the briquettes a used  
30       silica foundry sand, as described in PCT/EP95/02109. When it is desired to increase the content of  $\text{MgO}$  or  $\text{FeO}$ , olivine foundry sand may be used.

The mineral charge to the furnace may consist solely of the briquettes containing contaminated alumina sand or,  
35       more usually, is a mixture of at least 30%, often at least 50% or 60%, and typically up to 80% or more, of such briquettes with other particulate material. This other



material may comprise briquettes made from materials free of contaminated alumina sand and/or other mineral material suitable for incorporation in the melt. This additional mineral material may be industrial waste, as discussed  
 5 above, or may be virgin mineral material that has not previously been used.

Such mineral material, that can be used as part of the non-briquette charge or as components in briquettes, can include materials such as diabase, basalt, dumite,  
 10 peridotite, pyroxenite, apatite, bauxite, dolomite, iron ore, limestone, rutile, magnesite, magnetite, brucite, burnt lime, slag and other materials suitable for forming a fibre-forming melt. The blend of contaminated alumina sand, other industrial waste and other mineral material  
 15 should be such that the melt and the fibres have the desired composition.

The preferred composition for the total melt and for the fibres formed from it include, by weight of oxides,

	SiO <sub>2</sub>	32 to 48%
20	Al <sub>2</sub> O <sub>3</sub>	14 to 38%
	CaO	10 to 30%
	MgO	2 to 20%
	FeO	2 to 15%
	Na <sub>2</sub> O + K <sub>2</sub> O	0 to 12%
25	TiO <sub>2</sub>	0 to 6%
	Other Elements	0 to 15%

Other elements that can be present include P<sub>2</sub>O<sub>5</sub> and B<sub>2</sub>O<sub>3</sub>.

Preferably the amount of Al<sub>2</sub>O<sub>3</sub> is at least 16%, most preferably at least 17%, especially at least 18%. Usually  
 30 it is above 19%, e.g., 20 to 30%. The amount of MgO is usually 2 to 15% and the amount of Fe is usually 5 to 9%. The amount of Na<sub>2</sub>O + K<sub>2</sub>O is usually 0-6% and the amount of TiO<sub>2</sub> 0-3%. The total amount of the unspecified other  
 35 element is usually 0-5%.

Preferred fibres have a dissolution rate at pH 4.5 of at least 20 nm per day and the melt preferably has a viscosity at 1400°C of 10 to 70 poise.

Preferred fibres are those which are described and defined in WO96/14454 or in WO96/14274. The method for determining the dissolution rate is defined in those applications. Fibres having an analysis as in WO96/14454 or 96/14274 but wherein the amount of  $Al_2O_3$  is above 30%, e.g., 31 to 38% preferably have dissolution and melt properties as defined in WO96/14454 or 96/14274.

The briquettes may be made by any suitable method. Generally they are made by bonding inorganic material using a binder, often associated with compression of these briquettes. The binder can be a hydraulic binder such as cement or may be a slag activated with an alkaline agent, as in WO92/04289. The binder may comprise a burnt lime which is hydrated on heating in the presence of water, for instance as in the well known lime/sandstone process. The binder may comprise an organic binder, preferably molasses, optionally with burnt lime and usually also with fibres, as described in WO95/34514. The binder may comprise clay.

The briquettes may have conventional dimensions, for instance a minimum dimension of at least 5mm, often at least 20mm and usually at least 40mm and a maximum dimension of up to 300mm but usually not more than around 150 or 200mm.

The furnace may be heated in conventional manner, for instance as an electrical furnace or a tank furnace or, more usually by combustion of combustible material in a cupola furnace. The melt temperature will depend upon the minerals being used and the fibre-forming technique but is generally in the range 1200 to 1600°C, often around 1400 to 1550°C.

Fibre formation can be by conventional techniques such as a spinning cup technique or, preferably, by pouring on to a spinner comprising at least two co-operating spinning wheels, for instance as described in WO92/06047. Thus the

fibres may be made by pouring the melt on to a first spinning rotor from which the melt is thrown in sequence on to one or more subsequent spinning rotors off which the fibres are thrown.

- 5       The products of the invention can be used for any of the conventional uses of MMV fibres, such as thermal insulation, noise reduction and regulation, fire protection, growth media, reinforcement and fillers.

The following are some examples of the invention:

10   Example 1

Charge: 50% diabase + 45% Cement briquette

- Cement Briquette:

- 13% cement
- 35% wool waste
- 15   - 12% LD converter slag
- 30% Bauxite foundry sand
- 5% Olivine foundry sand
- 5% power plant bottom ash

Charge Chemistry (wt %):

20	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	FeO	CaO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	MnO	P <sub>2</sub> O <sub>5</sub>
	37.8	24.9	2.2	6.7	15.8	9.2	1.7	1.1	0.2	0.1

Melt viscosity at 1400°C: 23.5 Poise.

Example 2

Charge: 60% diabase + 10% LD converter slag + 35%  
25   briquettes.

- Briquette composition

- 7.5% Molasses
- 2.5% Burnt lime
- 10% Olivine foundry sand
- 30   - 35% wool waste
- 5% LD converter slag
- 40% Chamotte foundry sand

Charge Chemistry (wt %):

35	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	FeO	CaO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	MnO	P <sub>2</sub> O <sub>5</sub>
	43.8	16.4	2.5	8.3	14.8	10.8	1.8	1.1	0.3	0.2

Melt viscosity at 1400 °C: 18.6 Poise

Example 3

10

Charge: 25% diabase - 5% LD converter slag + 70% Cement briquette.

- Cement briquette:

- 13% Cement
- 5 - 37% Mullite foundry sand
- 18% Olivine foundry
- 22% Wool waste
- 10% LD converter slag

Charge Chemistry (wt %):

10	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	FeO	CaO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	MnO	P <sub>2</sub> O <sub>5</sub>
	33.5	28.8	1.0	5.7	17.5	11.5	1.0	0.5	0.3	0.3

Melt viscosity at 1400°C: 17.2 Poise

#### Example 4

15 Charge: 20% diabase + 20% LD converter slag + 60% briquettes.

- Briquette composition:

- 7.5% Molasses
- 2.5% Burnt lime
- 30% Wool waste
- 20 - 10% Olivine foundry sand
- 50% Kaoline foundry material

Charge Chemistry (wt %):

25	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	FeO	CaO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	MnO	P <sub>2</sub> O <sub>5</sub>
	40.6	19.0	1.2	7.8	19.2	8.8	1.8	0.5	0.5	0.5

Melt viscosity at 1400°C: 16.3 Poise

#### Example 5

Charge: 100% briquettes

- Briquette composition:

- 29 wt% Bauxite foundry sand
- 30 - 16 wt% Foundry quartz sand
- 10 wt% Iron oxide scale
- 18 wt% Ladle slag
- 9 wt% Cement
- 18 wt% Olivine foundry sand

## Charge Chemistry (wt %):

SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	FeO	CaO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	MnO	Others
30.5	33.3	1.2	9.3	13.7	10.3	0.1	0.3	0.6	0.8

Example 6

- 5 Charge: 100% briquettes
- Briquette composition:
    - 18 wt% Bauxite foundry sand
    - 12 wt% Foundry quartz sand
    - 16 wt% Ladle slag
    - 10 - 24 wt% Wool waste
    - 2 wt% Iron oxide scale
    - 12 wt% Molasses + Lime (8 + 4)
    - 16 wt% Converter slag

## Charge Chemistry (wt %):

SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	FeO	CaO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	MnO	Others
15 32.4	35.4	1.1	7.2	17.1	3.9	0.7	0.9	0.7	0.4

Example 7

- Charge: 92% briquette + 8% Karlshamn diabas
- Briquette composition:
    - 20 - 10 wt% Bauxite foundry sand
    - 22 wt% Foundry quartz sand
    - 22 wt% Wool waste
    - 2 wt% Iron oxide scale
    - 5 wt% Mg-spinel furnace lining
    - 25 - 10 wt% Cement
    - 23 wt% Olivine foundry sand
    - 6 wt% Glass cullet

## Charge Chemistry (wt %):

SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	FeO	CaO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	MnO	Others
30 48.5	14.2	0.7	6.3	10.3	17.3	1.4	0.8	0.1	0.3

CLAIMS

1. A method of making man made vitreous fibres comprising forming moulded briquettes of particulate mineral material, forming a melt by melting in a furnace a charge comprising the briquettes and forming fibres from the melt, characterised in that the melt and the fibres have a content, measured as oxides, that includes  $\text{Al}_2\text{O}_3$  in an amount of at least 14% by weight and the briquettes are formed of particulate material that includes used alumina sand contaminated with foundry residues.
2. A method according to claim 1 in which the contaminated alumina sand has an  $\text{Al}_2\text{O}_3$  content of at least 20% by weight.
3. A method according to claim 1 or claim 2 in which the amount of contaminated alumina sand in the briquettes is 10 to 75% based on the weight of the total mineral charge.
4. A method according to any preceding claim in which the fibres have the analysis including, by weight of oxides,
 

$\text{SiO}_2$	32 to 48%
$\text{Al}_2\text{O}_3$	14 to 38%
$\text{CaO}$	10 to 30%
$\text{MgO}$	2 to 20%
$\text{FeO}$	2 to 15%
$\text{Na}_2\text{O} + \text{K}_2\text{O}$	0 to 12%
$\text{TiO}_2$	0 to 6%
Other Elements	0 to 15%
5. A method according to any preceding claim in which the fibres have solubility at pH 4.5 of at least 20 nm per day.
6. A method according to any preceding claim in which the amount of  $\text{Al}_2\text{O}_3$  is at least 18%.
7. A method according to any preceding claim comprising the preliminary step of using the alumina sand as a foundry casting material.
8. A method according to any preceding claim in which the briquettes are formed additionally from other industrial waste selected from converter slag or other slag from steel making processes, glass, mineral fibre cement, power plant

ashes, wood ash, steel plant dust and man made vitreous fibre products, wherein the total amount of contaminated alumina sand and other industrial waste is at least 50% based on the total mineral charge.

- 5     9.    A method according to any preceding claim in which the furnace is a cupola furnace.
- 10    10.   A process according to any preceding claim in which the fibres are made by pouring the melt on to a first spinning rotor from which the melt is thrown in sequence on to one or more subsequent spinning rotors off which the fibres are thrown.

# INTERNATIONAL SEARCH REPORT

International application No.  
PCT/EP 96/05616

## A. CLASSIFICATION OF SUBJECT MATTER

IPC6: C03C 1/02 // C03C 13/06  
According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC6: C03B, C03C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

DIALOG: WPI, CLAIMS

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 9521799 A1 (ROCKWOOL INTERNATIONAL A/S), 17 August 1995 (17.08.95), page 7, line 24 - line 30, abstract  -----	1-10

☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

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SA 149882

## INTERNATIONAL SEARCH REPORT

Information on patent family members

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